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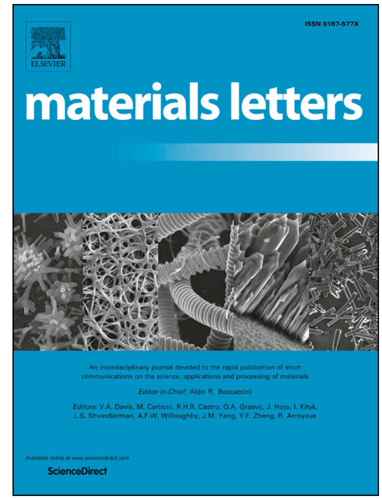

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Fractional factorial design to study admixtures used for 3D concrete printing applications

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Abstract

Mortar mix design for 3D printing applications is growing at a fast pace. The array of available materials proposed for such an application creates a lot of opportunities for mix designs. However, it makes the selection between the available options an onerous task to fulfill. The objective of this study is to reduce the material required to assess the importance of admixtures on fresh properties of cement-based mixes. Among the competing objectives required in a mix design for 3D printing applications, the dynamic yield stress and the flow are investigated. The dynamic yield stress of the cement-paste mixtures is measured by a rotary rheometer and the ASTM C1437 flow test is used to measure the spread of the mortar mixes. The spread is correlated with the pumpability of the mixes, whereas the dynamic yield stress indicates the extrudability and the structure deformation of the mixes. The cement used is blended with silica fume. Fine local sand is selected and the admixtures used are a superplasticizer based on synthetic organic polymers, a biopolymer polysaccharide viscosity modifying agent, a nanoclay made of magnesium silicate, crystalline calcium silicate hydrates and an accelerator with a water-reducing effect. Design of experiments methods are employed. To evaluate the influence of each admixture, the main effects and interactions are measured and the probability plots are graphically illustrating the results. First, an analysis is made on a two-level full factorial design with four factors. The results of the analysis are then compared with a half-fractional factorial design of a resolution IV. A fractional factorial design accurately discriminates the factors and reduce the number of experiments in mortar mix designs for 3D concrete printing applications.

Keywords

Mix design, Design of experiments, Admixtures, 3D concrete printing.

1. Introduction

Additive manufacturing has emerged in the construction field and 3D concrete printing is one of the methods. Many admixtures have been suggested to modify the rheology of mortar for 3D printing [1, 2], as well as many alternative binders and aggregates [3]. Those materials control the early stage properties, the final properties and simultaneously reduce the environmental impact of concrete. Two of the most important early age properties in this field are the flow and the yield stress. The spread of the mixes is correlated with the pumpability of the mixes [4]. High static and dynamic yield stress are advantageous for improving the resistance to structure deformation [5]. The pumpability and extrudability of the mixes depend on the extrusion system used, with a few examples in mortar mixes being a dynamic yield stress of 220 Pa and 386 Pa [5, 6], and a spread of 50-90% [4]. The Design of Experiments (DoE) is a tool to optimize the designing procedure and

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selecting the best samples in the design space [7]. Statistics and DoE are inextricably linked [8]. With DoE the designer can get the maximum amount of information using the minimum amount of resources [9]. The use of full-factorial design has been applied in the design of cement-based mixes for 3D printing applications [6]. However, the same results can be obtained by a fraction of the experiments needed in a full-factorial design. In this study an analysis was made initially on a 2^4 full factorial design to study four rheology modifiers and the results are compared with a 2^{4-1} fractional factorial design.

2. Materials and methods

The data for this study was obtained from [2]. Cement blended with silica fume and fine local sand were selected. The admixtures were a superplasticizer, a biopolymer polysaccharide viscosity modifying agent (B), nanoclay made of magnesium silicate (M), crystalline calcium silicate hydrates (C-CSH), and a accelerator with a water-reducing effect (A). Every mix contained the superplasticizer. The mix design was a 2-level full factorial design with 4 factors, and 16 mixes were needed to be tested. The 4 factors were the 4 out of 5 admixtures, meaning all except for the superplasticizer, and the two levels were their inclusion or exclusion from the mix [10]. A rotary rheometer was used to measure the dynamic yield stress of cement-paste mixtures and the ASTM C1437 flow test of mortar. The tests were conducted immediately after the mixing procedure. More details are found in [2]. A half-fractional factorial design with the same number of factors and levels would require only half of the experiments, meaning from 16 to 8. Respecting that those samples should be balanced and orthogonal [7], and having a $B \cdot M \cdot A \cdot CSH$ generator equal to +1 the selected samples would be the 1st, 4th,6th,7th,10th,11th,13th, and the 16th from those of the full factorial plan. Table 1 shows the full factorial mix design, the results that are analyzed with the probability plots and the selected mixes of the fractional factorial mix design marked in bold font.

Table 1. Two levels full factorial mix design with four factors (2^4) and their half-fractional factorial design (2^{4-1}) marked in bold font.

Mixes	*CSH*	*A*	*M*	*B*	*Cement paste Yield stress (Pa)*	*Mortar Flow (%)*
1	**-1**	**-1**	**-1**	**-1**	**9.7**	**106.0**
2	-1	-1	-1	+1	14.5	96.0
3	-1	-1	+1	-1	12.9	97.0
4	**-1**	**-1**	**+1**	**+1**	**18.2**	**85.0**
5	-1	+1	-1	-1	5.4	115.5
6	**-1**	**+1**	**-1**	**+1**	**8.4**	**110.0**
7	**-1**	**+1**	**+1**	**-1**	**5.8**	**107.0**
8	-1	+1	+1	+1	10.5	97.0
9	+1	-1	-1	-1	17.5	95.0
10	**+1**	**-1**	**-1**	**+1**	**25.6**	**92.5**
11	**+1**	**-1**	**+1**	**-1**	**23.2**	**92.0**
12	+1	-1	+1	+1	28.6	84.5
13	**+1**	**+1**	**-1**	**-1**	**9.0**	**95.5**
14	+1	+1	-1	+1	13.8	92.0
15	+1	+1	+1	-1	11.5	95.3
16	**+1**	**+1**	**+1**	**+1**	**14.1**	**75.5**

3. Results and discussion

3.1 Admixtures impacting the dynamic yield stress

After calculating the main effects and interactions, the analysis in the full factorial plan showed that the P had the biggest influence in the yield stress of the mix, followed by the addition of C-CSH and the addition of B. However, this specific type of A tested in these conditions had a negative impact, whereas the other two admixtures had a positive impact on yield stress. Specifically, the effect of the P was -8.9 Pa, the effect of C-CSH was +7.2 Pa and the effect of B was +4.8 Pa. In the probability plots, the filled and empty markers differ between statistically significant and statistically insignificant effects (Figure 1). Since the property of the material investigated is the dynamic yield stress, the estimated impact of each factor and interaction is in Pa.

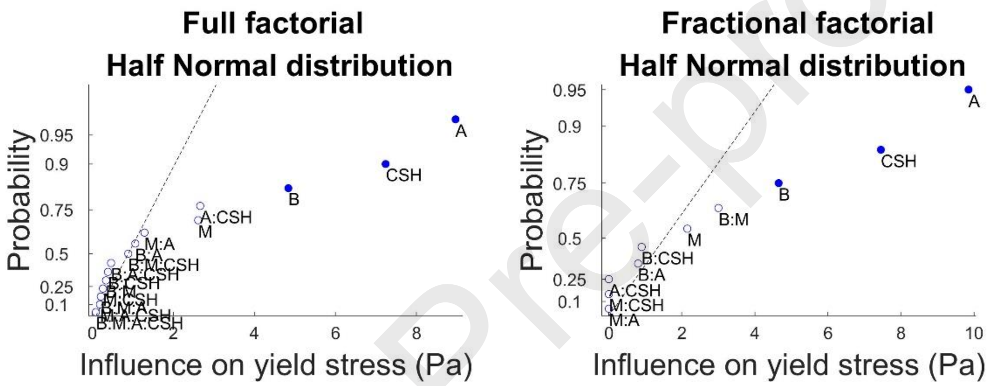

Fig. 1. The probability plot for the full factorial (a) and the fractional factorial design (b) of the dynamic yield stress of cement paste.

Making the same analysis in the half-fractional factorial design, the results were almost identical. Specifically, the three most important factors were the A, the C-CSH and the addition of B, with -9.85 Pa, +7.45 Pa and +4.65 Pa respectively.

3.2 Flow of mortar

The same analysis was made for the flow of mortar mixes (Figure 2). Since the property of the material investigated is the flow of the mortar, the estimated impact of each factor and interaction is the spread in the percentage of the initial diameter of the specimen. This time, the analysis in the full factorial plan showed that the addition of C-CSH had the biggest influence in the flow of the mix, followed by the addition of B and the addition of M. All of them have negative effects, meaning that the flow is reduced. Specifically, the effect of the C-CSH was -11.4 %, the effect of B was -8.85 % and the effect of M was -8.65 %.

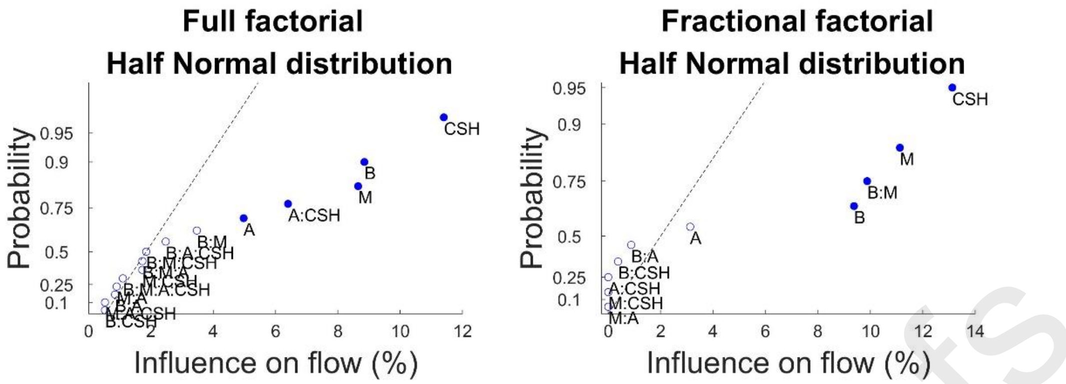

Fig. 2. The probability plot for the full factorial (a) and the fractional factorial design (b) of the flow of mortar.

The analysis in the half-fractional factorial design gave almost the same results. Specifically, the three most important effects were the effect of the C-CSH, followed by the M and the B, with -13.13 %, -11.13 % and -9.38 % respectively. The interaction effect of the B and the M was also an important effect having -9.88 %.

4. Conclusion

This brief investigation revealed that, of the four admixtures used, the addition of crystalline calcium silicate hydrate (C-CSH) was the most significant in terms of dynamic yield stress and flow, as assessed immediately after the mixing procedure. This specific accelerator (A) gave the adverse results due to its water-reducing effect. The biopolymer polysaccharide viscosity modifying agent (B) had better influence at the dynamic yield stress than the nanoclay made of magnesium silicate (M). The same conclusions were drawn after following a full factorial and a fractional factorial plan, promising that half of the experiments could be avoided, reducing the amount of resources needed. Hence, for future studies, the A and M admixtures will be eliminated to reduce the number of factors of the design and a fractional factorial plan will be preferred. They can be selected with other conditions.

Acknowledgement

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Highlights:

The methodology reduces the material required to assess the importance of admixtures.

The same conclusions were drawn following a full and a fractional factorial plan.

Half of the experiments could be avoided, reducing the amount of resources needed.

Credit author statement

Vasilis Sergis: Conceptualization, methodology, investigation, data analysis, writing original draft and review, visualization

Claudiane Ouellet-Plamondon: Conceptualization, methodology, data curation, writing review and editing, funding acquisition

Declaration of Interest Statement

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